PATENT SPECIFICATION

Application Date: Feb. 6, 1933. No. 3568/33.

403,239

Complete Accepted: Dec. 21, 1933.

COMPLETE SPECIFICATION.

Improvements in the Treatment of Natural and Artificial Silk.

I, EDWIN CHARLES AXE, A.I.M.E., a British subject, of 27, Chancery Lane, London, W.C. 2, do hereby declare the nature of this invention (a communication to me from Maywood Chemical Works, a Corporation organised under the laws of New Jersey, United States of America, of Maywood, New Jersey, United States of America), and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

The primary object of this invention is to provide a way by which both natural and artificial silk may be quickly weighted with less labour, less danger to the operator, and with less injury to the produced goods than with existing processes. By the term "artificial silk" there is understood any fibre which either entirely or partly consists of cellulose regenerated from a cellulose solution, of a cellulose ester or ethers.

The corrosiveness of tin-tetrachloride solution to the hands of operators and to the utensils needed in the weighting process, and the destruction which the deposited tin compounds gradually exerts upon the silk fibre have combined to present a problem causing consumers to make efforts to overcome these drawbacks of tin-tetrachloride solution.

Theoretically tin-tetrachloride is a neutral substance, but actually it displays in its various applications a rather acid character. Refer, for example, to "Artificial silk" (Hottenroth, 1928, page 369).

In carrying out the primary object aforesaid, the present invention provides a process for weighting natural and artificial silk with tin-tetrachloride to overcome the harmful acid effect. It has been found by my communicants that tin-tetrachloride solutions de-acidified with carbonates of metals or mixtures of same which are characterised by forming a chloride soluble in water and a phosphate which is insoluble therein, for example lithium, barium, calcium and so forth as specified later herein, including carbonates of the rare earth metals, do not deleteriously affect natural and artificial silk, whilst they cause the same to take up more weight when subsequently washed with water during the weighting process.

The following equation illustrates the de-acidification:

$$3SnCl_4 + La_2(CO_3)_3 + 6H_2O = 3Sn(OH)Cl_3 + La_2(OH)_3Cl_3 + 3H_2CO_3$$

When treated with the mixture of basic metal chlorides thus obtained in aqueous solution, natural and artificial silk becomes more heavily weighted than when treated under the same conditions with a plain tin-tetrachloride solution, and then with solutions of other metal salts such as alkali metal silicates, phosphates, tungstates or other similarly acting salts.

It is found that the presence of the said other basic metal chlorides accelerates and favours the precipitation of the tin-oxyhydrate upon both natural and artificial silk.

The prime requirements of thus de-acidified aqueous tin-tetrachloride solution is that the solution shall be clear and colourless, and remain free of sediments or precipitates. It has been found that this requirement is fulfilled by solutions de-acidified by all metal carbonates, the chlorides and phosphates of which fulfil the aforesaid conditions of solubility.

The solutions of the character pointed out above when applied to artificial silk render the same absorptive enough to be as highly weighted as natural silk.

High atomic weight is not so much a determining factor, but rather the readiness of added carbonates dissolving in the concentrated tin-tetrachloride solution to form a stable solution of great durability and high specific gravity.

The carbonates of magnesium and zinc are especially suitable for the preparation of strongly basic and highly durable colloidal tin chloride solution. Silk and artificial silk weighted and phosphated by the present process do not incline to deterioration in sunlight.

Carbonates of other metals applicable for the present process are carbonates of lithium, barium, calcium, magnesium, strontium, cadmium, copper, iron, chromium, uranium, lead, bismuth, zirconium, titanium, alone or in mixture.

[Price 1/-]

The carbonates of the aforesaid metals may also be used in association with carbonates of one or more of rare earth metals e.g. cerium, lanthanum, didymium, thorium, scandium, yttrium, praseodymium, neodymium, samarium, terbium, europium, gadolinium, ytterbium and other representatives of the group.

The obtained solution is allowed to settle the undissolved particles the clear part is removed, whereupon the solution is ready for immediate application.

EXAMPLE.

Introduce 100 kgs, metal carbonate, the chloride of which is soluble and the phosphate insoluble, into 750 kgs. of 50 Bé tin-tetrachloride solution, at 75° C. (for instance 100 kgs. zinc carbonate introduced) and the solution ultimately obtained gives practically a neutral reaction.

Silk (first degummed) or artificial silk, such as made of viscose, is allowed to soak for some time, for instance one and one half hours, in the yielded solution of basic zinc tin chloride; it is then taken out, the surplus of solution squeezed out and the silk or viscose washed with a little water. The washed silk or viscose is then introduced into a solution of metal salt giving with the tin compound in the fibre an insoluble salt. Such solution for instance containing alkali-metal phosphate—e.g. di-sodium phosphate of 70° C.

The silk or viscose is allowed to hang in the phosphate one and one half hours, is then washed and dried. This procedure may be repeated as often as desired.

To indicate the total increase in weight with weighting solutions containing mixed basic chlorides of various metals in comparison with the treatment with tin-tetrachloride only the alternate soaking in each weighting solution and in the phosphating bath was operated altogether three times to put the examples on the same basis.

Three alternate passages through the weighting solution and phosphating solution give, for silk, with plain tin-tetrachloride total increase in weight 51.14%, with basic tin lead chloride 60%, basic tin zinc chloride 66.3%, basic tin-zirconium chloride 68.47%. basic neodymium magnesium tin chloride 72.7% for viscose, with basic tin zinc chloride 84.34%, with basic tin magnesium chloride 86.66%.

The carbonates of metals are substitutable by hydroxides of other weighting metals—e.g., of zinc, magnesium, aluminium, tin, rare earth metals, and alkali salts in which the weighting metal, contained therein, is in the anion radicle, for example, sodium zincate, sodium aluminate, sodium plumbate, sodium stannate.

Sodium phosphate is substitutable by silicate, tungstate, molybdate and other known heavy salts used in weighting.

The weighting process is very simple and is operated without much scientific control whilst the poisonous character here may be disregarded.

Tin tetrachloride for de-acidification is substitutable by aluminium chloride, iron chloride, chromium chloride, manganese chloride, zirconium chloride, titanium chloride, other chlorides of known weighting metals. Obviously some metals have a double function, i.e. as a weighting metal, and as a de-acidifying compound, but the weighting solution always contains the basic chlorides of at least two metals.

Having now particularly described and ascertained the nature of my said invention, and in what manner the same is to be performed, I declare that what I claim is:—

1. A process of weighting both natural and artificial silk fibre, consisting in acting on the fibre with a colloidal aqueous solution of basic chlorides prepared by the addition, to a solution of a chloride of a weighting metal, of one or more carbonates or hydroxides of other weighting metals or of one or more alkali salts containing a weighting metal in the anion radicle, the said weighting metals being such that their chlorides are soluble and their phosphates are insoluble, washing the fibre with water, and subjecting the fibre thus treated to the solution of a salt capable of forming an insoluble salt with the metals of the salts of the aforesaid aqueous solution.

2. A process of weighting both natural and artificial silk fibre according to claim 1, wherein the chloride of the weighting metal, to the solution of which the other substance or substances containing a weighting metal is added, is tin tetrachloride.

Dated this 6th day of February, 1933.

EDWIN C. AXE, A.I.M.E.,
27, Chancery Lane, London, W.C. 2.

Redhill: Printed for His Majesty's Stationery Office, by Love & Malcomson, Ltd.—1934.